Fabrication and mechanical characterisation of carbon fabric reinforced epoxy with alumina and molybdenum disulfide fillers

Bheemappa Suresha*
Department of Mechanical Engineering,
The National Institute of Engineering,
Mysuru – 570008, India
Email: sureshab@nie.ac.in
Email: sureshab2004@yahoo.co.in

Manpinder Singh Saini
Engineering Services Department,
Infosys Limited,
Mysuru – 570027, India
Fax: +91-821-2485802
Email: manpinder.singh28@gmail.com

Abstract: In this research article, a systematic study has been carried out to investigate the mechanical properties of carbon fabric reinforced epoxy (C-E) composites, having alumina (Al₂O₃) in one instance and molybdenum disulfide (MoS₂) of two different loadings in the epoxy matrix resin. The study has revealed that with 60 wt.% carbon fabric loading brings superior mechanical properties to the epoxy matrix. The micron-phased (Al₂O₃/MoS₂ in epoxy resin) matrix is then utilised with T300 carbon fabric performs to fabricate hybrid laminated composites. The resulting structural hybrid composites have been tested under tensile and flexural loads to evaluate mechanical properties. The fillers were micron-sized Al₂O₃/MoS₂ particles which were mixed with epoxy resin using high shear mixer. The amount of particle loading varied in two steps viz. 5% and 10% by weight. It has been observed that microparticles inclusion increases the thermal stability of the system by enhancing cross-linking in the epoxy matrix. Microparticles also tend to reduce air void content of the as-fabricated hybrid composites and thus translate into increased mechanical properties. With 10 wt.% loading of MoS₂ in C-E enhanced the flexural strength and modulus by 20 and 40% respectively.

Keywords: carbon fabric reinforced epoxy; microfillers; mechanical properties.


Biographical notes: Bheemappa Suresha is a Professor of the Department of Mechanical Engineering, The National Institute of Engineering, Mysuru since 1990 and is currently the Head, Centre for Composite Materials Research. He obtained his Diploma (1983) in ME from CPC Polytechnic, and obtained his BE degree from NIE, Mysuru (1987) affiliated to UoM Mysuru, MTEch in
1 Introduction

Polymers and their composites are emerging as possible alternative products to metal-based composites in many common and advanced engineering applications. The ease of fabrication, the availability of a good choice of materials from both thermoplastic and thermoset varieties and economic viability have made the advent of these newer materials for industries ranging from automobile to sports goods. Development of large structural laminates from micron-phased polymer composites showed good mechanical properties (Mallik, 2007; Nakamura et al., 1992; Zhang et al., 2004). With our continuing search for lighter and stronger polymer-based composites, the demand for new types of materials is increasing. The fibres play an important role on the load carrying capacity along the fibre direction, endowing the composites with superior in-plane performance. Woven fabrics are produced by interlacing of warp fibres and weft fibres in a regular pattern or weave style. The integrity of fabrics is attributed to the mechanical interlocking of the fibres. A number of studies in particulate filled polymer composites have been undertaken with microparticles (Nakamura et al., 1992; Zhang et al., 2004; Kolling et al., 2003). Many investigations in the field of polymer matrix composites (PMCs) are either short or continuous fibres. Woven fabric composites are extensively used because of their balanced properties in the plane of the fabric and ease of handling during fabrication (Vishwanath et al., 1991).

Mechanical properties of PMCs depend on the type of fabric, matrix, type of weave, surface treatments on fabric and particulate fillers of micro or nano-size. Studies developed by Vishwanath et al. (1993) reported that glass fibre (GF) reinforced poly vinyl butyral (PVB) modified phenolic composites was increased the tensile, flexural, impact and interlaminar shear strength (ILSS). In research work involving glass/carbon fabric reinforced vinyl ester composites, the tensile strength of carbon-vinyl ester composite is higher than that of glass-vinyl ester composite. Results of this study indicated that the high strength carbon fibre reinforcement was responsible for the
Fabrication and mechanical characterisation of carbon fabric

Improved tensile strength of vinyl ester composite (Suresha and Kumar, 2009). The influence of different weave forms on mechanical properties was investigated by Bijwe and Rattan (2007) by taking three composites of polyetherimide (PEI) containing CF with three weaves, viz. plain, twill and satin and concluded that twill weave was the best from the stand point of tensile strength, tensile modulus, flexural strength, flexural modulus and ILSS, but not the toughness.

The performance of fibre reinforced composite materials was always related to the interfacial bond strength that requires special tests, fixtures, and materials for measuring it. Recently, the mechanical and tribological properties of micron-sized inorganic fillers were studied in several cases (Suresha et al., 2007a, 2007b, 2007c, 2008; Kumaresan et al., 2011). The present research trend is to enhance the mechanical properties by the incorporation of particulate filler to the fabric reinforced polymer composites. The inclusion of fillers like SiC, SiO2, graphite and Al2O3 in woven glass fibre reinforced epoxy composites have been investigated. Fillers like SiC, SiO2 and graphite in glass fibre reinforced epoxy composites showed increasing trend of mechanical properties with increasing filler content. Hussain et al. (1996) have investigated the effect of alumina particle size (1 µm and 25 nm) on the mechanical properties of C-E composites. They reported that the hybridisation of the carbon fibre reinforced epoxy composites by Al2O3 micro/nano-particles showed significant improvements in the mechanical properties. It has been established in recent years that polymer-based composites reinforced with a small percentages of strong fillers can significantly improve the mechanical, thermal and barrier properties of the pure polymer matrix (Hlangothi et al., 2003; Walter et al., 1997).

In the present work, micron-sized Al2O3/MoS2 particles have been dispersed through high shear mixer with a room temperature cure epoxy matrix. The modified resin was then used to reinforce the carbon fabric for preparation of hybrid microcomposites. These composites characterised for their mechanical properties. Since fine microparticles (< 25 µm) incorporation was seen to be more promising than the bigger microparticles inclusion, fine micron-phased matrix was chosen to fabricate laminated composites using bi-directional carbon fabric performs. The resulting laminated hybrid composites were characterised under static loading. Details of the manufacturing procedure and analyses of test results are described in the following sections.

2 Experimental details

2.1 Materials and sample preparation

The composite materials considered in the present investigation consists of bi-directional carbon fabric of about 6–8 µm diameter as reinforcement. LY 556 epoxy resin with HY951 grade room temperature curing hardener with diluents DY 021 (all supplied by Hindustan Ciba Geigy) mix was used for the matrix material. The filler materials used (Al2O3 and MoS2) are of particle size ranging from 20 to 25 µm. The fillers Al2O3 and MoS2 were purchased from Sigma Aldrich and Tianjin Okeyon International Trade Co. Ltd respectively. These fillers were treated with 2% organo-reactive silane coupling agent.
Pre-calculated amount of epoxy resin and Al\textsubscript{2}O\textsubscript{3}/MoS\textsubscript{2} filler were mixed using high shear mixture (T-T18 ULTRATURRAX Basic) at an operating speed of 2,000 rpm for 10 min. The temperature during mixing was maintained at 50°C. Eight layers of fabric were prepared to obtain 3 mm thick laminates. The carbon epoxy composite were prepared by hand layup procedure followed by compression moulding. The panels have been cured using auto clave facility (pressure 7.35 MPa and temperature 390°C). Table 1 shows the details in respect of designation and weight percentage of epoxy and micron-sized fillers in 60 wt.% carbon fabric reinforcement used in the present investigation.

Table 1  Composites fabricated for present work

<table>
<thead>
<tr>
<th>Composites (designation)</th>
<th>Epoxy (wt.%)</th>
<th>Al\textsubscript{2}O\textsubscript{3} (wt.%)</th>
<th>MoS\textsubscript{2} (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon-epoxy (C-E)</td>
<td>40</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Al\textsubscript{2}O\textsubscript{3} + CE (CE-5A)</td>
<td>35</td>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>Al\textsubscript{2}O\textsubscript{3} + CE (CE-10A)</td>
<td>30</td>
<td>10</td>
<td>-</td>
</tr>
<tr>
<td>MoS\textsubscript{2} + CE (CE-5M)</td>
<td>35</td>
<td>-</td>
<td>5</td>
</tr>
<tr>
<td>MoS\textsubscript{2} + CE (CE-10M)</td>
<td>30</td>
<td>-</td>
<td>10</td>
</tr>
</tbody>
</table>

2.2 Determination of voids

Void fraction is a measure of the empty spaces in a material, and is a fraction of the volume of voids over the total volume. There are many ways to determine the voids in the composites. In the present work, for the composite specimens prepared, the volume fraction of voids was found from the following equation:

\[ V_v = \left( \frac{\rho_t - \rho_m}{\rho_t} \right) \times 100 \]  \hspace{1cm} (1)

where \( V_v \) is the void volume in percentage, \( \rho_t \) in g/cm\textsuperscript{3} is the theoretical density of the composite specimen and \( \rho_m \) g/cm\textsuperscript{3} is the measured density of the composite specimen respectively.

2.3 Barcol hardness

The Barcol hardness test characterises the indentation hardness through the depth of penetration of an indenter, loaded on a material sample and compared to the penetration in a reference material. The governing standard for the Barcol hardness test is as per ASTM: D-2538-13a. At least three specimens of each composition were tested and the mean values are shown in Figure 1.
2.4 Tensile testing

The tensile strength and the Young’s modulus of carbon-epoxy composite with and without fillers were determined according to ASTM D 638 M-93. The dog-bone-shaped test specimens were cut out from the composite laminates. The specimens were tested at a crosshead speed of 2 mm/min. At least five specimens for each composition were tested.

2.5 Flexural testing

Bending test was applied to carbon-epoxy composite with and without fillers in order to determine the flexural properties. ASTM D790M-86 was followed to determine the bending strength and the modulus of elasticity for both unfilled and particulate filled carbon-epoxy composites. Test Method 1 of ASTM D790M-86 was used in which a three-point loading system-utilising centre loading on a simply supported beam is employed. Specimens were machined from laminates and tested with a crosshead speed of 1 mm/min. The test specimen had dimensions of 90 mm × 12 mm × 3 mm. Five specimens from each set were tested.

The tensile and bending tests were carried out on a fully automated Kalpak-100K universal testing machine connected to a computer which was aided by KALPAK software. A 50-kN load cell was used.
3 Results and discussion

3.1 Density and volume fraction

Density of a material plays an important role in various weight sensitive applications. Density of PMCs depends on the relative proportion of the matrix and reinforcing materials. Based on the rule of mixtures and proportion of the constituent materials used in PMCs one can determine the theoretical density of the composite. Due to the presence of voids in a composite, which creeps in during the fabrication process, there is always a difference in the experimentally measured and the theoretical density value calculated from weight fraction. The void content of PMCs may significantly affect the mechanical properties. Higher void content of a composite lowers the fatigue resistance, greater susceptibility to water penetration and weathering (ASTM D2734). Hence, the knowledge of void content is useful in the assessment of quality of PMCs.

The theoretical and measured densities of the particulate filled CE composites under investigation, accompanied with the corresponding volume fraction of voids are as listed in Table 2. It is found that the theoretical and experimentally measured densities are not equal. Also theoretical densities are slightly higher than experimentally measured densities due to the presence of voids. The volume fraction of voids in Al2O3 filled C-E composites are slightly higher than the other composite samples investigated. In all the composites considered for investigation, the volume fractions of voids are very less (< 3%) which may be due to the fabrication process involved in the manufacture of these composites.

<table>
<thead>
<tr>
<th>Composites</th>
<th>Theoretical density (g/cm³)</th>
<th>Measured density (g/cm³)</th>
<th>Volume fraction of voids (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-E</td>
<td>1.432</td>
<td>1.412</td>
<td>1.396</td>
</tr>
<tr>
<td>CE-5A</td>
<td>1.485</td>
<td>1.441</td>
<td>2.962</td>
</tr>
<tr>
<td>CE-10A</td>
<td>1.551</td>
<td>1.506</td>
<td>2.901</td>
</tr>
<tr>
<td>CE-5M</td>
<td>1.508</td>
<td>1.492</td>
<td>1.061</td>
</tr>
<tr>
<td>CE-10M</td>
<td>1.590</td>
<td>1.562</td>
<td>1.761</td>
</tr>
</tbody>
</table>

3.2 Barcol hardness

The property hardness is one of the prominent factors that influence the wear resistance of any material. In the present study, the Barcol hardness of the CE filled with Al2O3 and MoS2 particulate filled composite have been obtained along with the hardness of CE composite and plotted in Figure 1. The results indicate that the hardness of particulate filled CE composite and this improvement is due to the addition of filler content. Further, the increase in hardness increased with filler loading which clearly indicating that improvement in hardness is a function of filler loading. On comparing Barcol hardness and density of the particulate filled CE composites, one can conclude that the hardness is roughly correlative with density. The closer packing of atoms would give greater density and would allow shorter bond lengths, which gives rise to greater hardness. Thus, MoS2 filled CE composites have marginally improved Barcol hardness compared to that of Al2O3 CE composites.
3.3 Tensile properties

The experimental data of tensile strength of particulate filled CE composites under investigation are recorded and plotted in Figures 2 and 3 respectively. The effect of filler loading on the tensile strength behaviour is shown in Figure 2. From the figure, it is observed that the tensile strength of Al₂O₃ and MoS₂ filled CE composites increase with increase in filler content. Among the two particulate fillers used in the investigation, the addition of MoS₂ result in maximum increment in tensile strength compared to Al₂O₃ filled and un-filled CE composites. The increase in tensile strength of particulate filled C-E over unfilled C-E composites can be due to the enhanced interface bonding of the silane treated filler particles and the matrix, which transfer the applied load effectively to the particulates. The interface bond strength depended on the effectiveness of the coupling agents and the inherent wetting ability of the polymer.

The tensile strength of MoS₂ filled is higher than Al₂O₃ filled C-E composites for two different filler loadings. The MoS₂ particulate filled C-E composite has more interfacial adhesion than that of Al₂O₃ filled composite which is evident from the fact that the contact angle for MoS₂ filled is less (860) than Al₂O₃ (1,000) filled C-E composite. This behaviour may also be attributed to high strength and hardness of the particulates used in research work. This behaviour is also attributed to the presence of slightly higher volume fraction of voids present in Al₂O₃ than MoS₂ filled C-E composite. The experimentally measured values of tensile modulus of different composites are plotted in Figure 3. The tensile modulus increases with the increase in filler loading for both Al₂O₃ and MoS₂ filled C-E composites. The tensile modulus can be improved by adding particulate fillers to a polymer matrix since hard particles have much higher stiffness than the matrix (Fu et al., 2008). Earlier research reports demonstrated an increase in filler loading improves the tensile modulus of the filled PMCs (Amdouni et al., 1992; Dekkers and Heikens, 1983; Wang et al., 1998). The tensile modulus of MoS₂ filled C-E composite is higher than that of Al₂O₃ filled CE and may be due to better interfacial bonding of particulates with the matrix.

Figure 2  Variation of tensile strength for different filler type and loading (see online version for colours)
3.4 Fractography of tensile test failed composites

Tensile fractured surfaces were examined by scanning electron microscopy (SEM). Fractured surfaces of unfilled C-E, Al₂O₃ filled and MoS₂ filled C-E hybrid composites are shown in Figures 4(a), 4(b) and 4(c) respectively. For unfilled C-E, it can be observed that extensive matrix damage takes place during tensile testing. Few river marks are evident from the micrograph [Figure 4(a)]. The river marks are steps between cleavage lines, which are always expected to propagate in the multi direction from the point of crack initiation [Figure 4(a)].

For Al₂O₃ filled C-E composite, few fibre pull-out and better adhesion of Al₂O₃ into the epoxy matrix are evident from the SEM picture show in Figure 4(b). Under the tensile loading conditions, most particles do not fracture as shown in Figure 4(b), which is obtained on the tensile fracture surface of Al₂O₃ filled C-E specimen. As shown in Figure 4(b), several mechanisms can be identified, including matrix cracks, particles pull-out, debonding of filler-matrix and a significant step appearance of fiber pulled-out region.

Figure 4(c) shows the tensile fractured surface of MoS₂ filled C-E composite. From the figure it can be observed that the epoxy matrix failure starts from weaker interface. Step structures showing fracture in different planes are formed behind fibres due to good bonding. The presence of fibres contributes to the formation of additional step structures from which new surfaces were formed. The failure mechanisms were identified as being: debonding and fibre fracture, pull-out of few fibres from the matrix and cross sectional fibre fracture.
3.5 Flexural properties

The results of flexural strength and flexural modulus of unfilled and particulate filled CE composites are shown in Figures 5 and 6, respectively. The results indicated that Al₂O₃ and MoS₂ fillers have significant effect on flexural strength and flexural modulus of CE composites. The flexural strength and modulus of 10 wt.% Al₂O₃ filled CE changed respectively by 14% and 17% compared to that of unfilled CE composite. However, the effect of MoS₂ filler loading is more significant and the enhancement in flexural strength and modulus were 21% and 40% for 10 wt.% loading in CE composite.
The effect of filler loading on the variation of flexural strength behaviour is shown in Figure 5. Both fillers showed improvement in the flexural strength of the CE composites. These particles roughening the fibre surface and provide strong bonding at the fibre/matrix interfaces that is caused by thermal residual stresses on the fibre surface. These roughness and strong interfacial adhesion act as mechanical interlocking that leads to improving the flexural strength of particulate filled CE composites.

The influence of filler loading on the flexural modulus is presented in Figure 6. From these it can be observed that flexural modulus was improved by incorporating Al₂O₃ and MoS₂ fillers in C-E composite. It is also evident that the addition of MoS₂ filler is more beneficial than that of Al₂O₃ filler in improving the flexural strength and modulus of CE composite. This result can be interpreted as follows. The flexural modulus is a material constant, which depend on the slope of the first linear portion of the load-deflection curve. The constituents of CE composite (carbon fibre and epoxy matrix) have the same elastic deformation in the load-deflection curve. On the other hand, high stiffness and high and strength of Al₂O₃ and MoS₂ particles under low loads cannot be synchronised with the deformation of fibre and matrix and accordingly, micro-cracks will be initiated through the matrix above or below the particles and propagated parallel to the fibre direction. Therefore, the modulus of unfilled is less than that of particulate filled CE composites. The improvement in the flexural strength and modulus of particulate filled CE composites were due to the enhancement in the interfacial bond strength between the micron-phased matrix and the carbon fibre.
4 Conclusions

Based on experimental observations of mechanical testing on CE and particulate filled CE composites, the following conclusions may be drawn.

1. There is a difference between theoretical and experimental density of the composites due to presence of voids. The volume fraction of voids in CE composite filled with Al₂O₃ is higher than unfilled and MoS₂ filled CE composite.

2. The wettability of MoS₂ filler in CE is better than that of unfilled and Al₂O₃ filled CE composites. Hence, interfacial adhesion of MoS₂ particulate filled CE composite is more than unfilled and Al₂O₃ filled CE composite.

3. The hardness of MoS₂ filled composite is higher than Al₂O₃ filled composite. This may be attributed due to the high density of MoS₂ filler.

4. The well dispersion of Al₂O₃ and MoS₂ in carbon fabric reinforced epoxy resin played a key role in improving the filler-matrix interfacial bond strength. Therefore, the applied stress was effectively transferred to the particles from the matrix and accordingly, the tensile strength, modulus, flexural strength, and modulus of MoS₂ filled CE composites are enhanced by 14%, 17%, 21% and 40%, respectively.
References


